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NRL Report 6050

Electrolytic Cell for X-Ray Diffraction Studies of Electrode Phenomena

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March 2, 1964



6491
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ABSTRACT

A

An electrolytic cell has been designed for use on a horizontal circle x-ray spectrogoniometer to permit simultaneous diffraction and electrochemical study of electrode specimens. This cell has as one electrode a partly submerged rotating disk of the sample from which x-ray diffraction patterns are made as a portion of this electrode rotates out of the solution. The cell is designed to study electrochemical processes of widely varied nature including active material transformations in primary and secondary battery electrodes, electrolytic deposition and corrosion phenomena. To illustrate the application of the cell to electrochemical investigations, preliminary work shows that silver oxides can be identified on a working silver electrode in KOH solution.

PROBLEM STATUS

This is an interim report and work on this problem is continuing.

AUTHORIZATION

NRL Problem C05-14
Projects RR001-01-43-4755 and SF013-06-03-4366

Manuscript submitted November 27, 1963.

ELECTROLYTIC CELL FOR X-RAY DIFFRACTION STUDIES OF ELECTRODE PHENOMENA

INTRODUCTION

The identification of materials taking part in an electrochemical reaction is a necessary part of characterizing fundamental electrode processes. Measurements of a purely electrochemical nature are not definitive of themselves and must be augmented by analytical data to identify the species acting in an electrode system. In many instances side reactions are possible, and it is of interest to know whether and under what conditions side reactions actually take place. When solid crystalline phases are involved, x-ray diffraction may be used for their identification. Usually the electrode is removed from the electrolytic cell for separate x-ray diffraction examination, however some investigators (1-5) have used electrolytic cells in place on x-ray machines under various conditions.

Briggs (1) used a wire electrode oscillating partly out of the electrolyte, registering the x-ray diffraction pattern from the upper part of the wire. When an unstable product was being formed, there could be self-discharge on the upper part of this wire, where the effective current density was lower than the average value for the wire as a whole (2). Falk (3) sealed a pair of electrodes in a thin polyethylene bag. Before x-ray examination, the surface of the test electrode facing the counter electrode was moved to the edge of the plastic to minimize electrolyte thickness. The resulting pattern included polyethylene peaks. Salkind and Bruins (4) used plastic cells with horizontal electrodes on a vertical circle x-ray goniometer. The test electrode did not face the counter electrode and the patterns had to be corrected since the surface being examined was not in the same plane as the rotation axis of the goniometer.

The NRL cell, designed for use on a General Electric horizontal circle x-ray spectrogoniometer, permits the x-ray examination to be made simultaneously with electrochemical measurements and visual observations. The cell has as one electrode a partially submerged rotating disk of a sample from which x-ray diffraction patterns are made as a portion of the electrode rotates out of the solution. This cell is designed to study electrochemical processes of a widely varied nature including active material transformations in primary or secondary battery electrodes, electrolytic deposition, and corrosion processes. The use of this type of cell greatly lowers the possibility of an unstable crystalline compound being present but undetected by x-ray diffraction, since the pattern may be registered at any time without interrupting the flow of current. Some preliminary work on the silver electrode in KOH solution illustrates the application of the cell to electrochemical investigations.

CELL DESIGN

The cell, shown in Fig. 1, has all parts made of Teflon except the screws, the pulley, and the electrical contact system. An electrode of the specimen material, a disk 57 mm in diameter and any thickness up to 6 mm, is mounted vertically in the cell so that it is always partially submerged in electrolyte. The specimen disk is held tightly against platinum contact wires by a threaded retainer ring. The inner edge of the specimen disk can be coated with an inert stopcock grease to prevent any electrolyte that might leak past the retainer from reaching the back side of the sample. Platinum contact wires

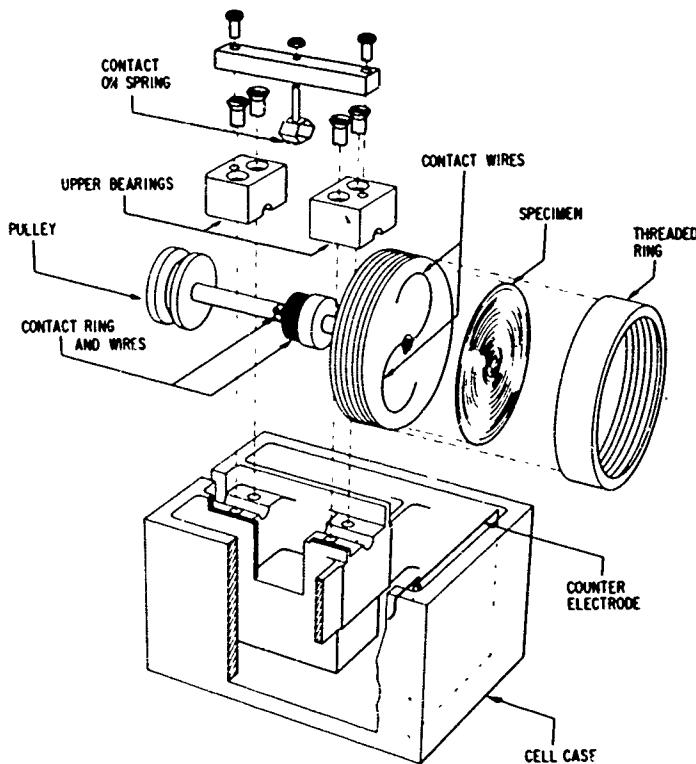


Fig. 1 - Expanded cutout view of electrolytic cell

pass through a hole in the drive shaft and are connected to the contact ring by a small screw. This contact ring is machined from brass and plated consecutively with layers of nickel-cobalt, gold, and finally rhodium to prevent corrosion. The moving contact is made through two small buttons of Palinay which are fastened onto a gold-plated beryllium-copper spring.

The horizontal x-ray beam passes above the cell case and is reflected from the upper portion of the rotating electrode which is not submerged. A counter electrode faces the submerged part of the specimen disk in the same compartment of the cell. A reference electrode can be put in one of the other compartments where it does not interfere with the x-ray beam path.

A synchronous motor with appropriate reduction gears rotates the specimen disk by a belt drive to the pulley. The specimen rotates continuously and is always wet with a film of electrolyte. Because the electrode is always partially submerged, the current need not be interrupted during the x-ray examination. The rotation rate of the specimen disk may be varied so that the time elapsing between total submersion and registration of the x-ray pattern may be made quite short and is limited only by the onset of splash. At the rate of 10 rpm used in this preliminary work, the x-ray pattern is registered 1.5 seconds after the sample leaves the meniscus with a total drain time of 3 seconds.

On a spectrogoniometer, the pattern is recorded from only those crystals lying in the sample surface in the reflecting positions within the area covered by the impinging x-ray beam. In using this cell, a larger than normal area of the sample is scanned by the beam as different sectors move into position during registration of the patterns. This same principle is used in the flat sample spinners available for study of medium and large grained specimens. The success of the diffractometer technique depends upon having a sufficient number of crystals in the correct orientations in the sample surface to give satisfactory signal intensity. The rotating specimen will tend to insure maximum

intensity for the diffraction peaks because more crystals contribute to the diffracted rays. This increase will tend to offset the loss in intensity caused by adsorption of the beam in the thin film of the electrolyte coating the sample.

The cell is shown in place on the spectrogoniometer in Figs. 2 and 3. The cell is aligned on the spectrogoniometer by eye and the final adjustment may be conveniently achieved by optical means. The vertical rotating specimen and the spectrogoniometer comprise a two-circle goniometer and the alignment is performed in an analogous fashion. The cell mounting (Fig. 4) provides adjusting screws for bringing the sample surface into position in the horizontal plane. Shim stock may be used under the cell to bring the surface into vertical position. The axis of rotation of the horizontal circle must lie in the sample surface and this is readily attained by using a telescope with cross hairs to view the stationary sample as it is swung through the arc of the spectrogoniometer. When in correct position, the sample image lying on the cross hair will not move during rotation of the spectrogoniometer. Satisfactory diffraction patterns may usually be obtained from the specimen after this adjustment.

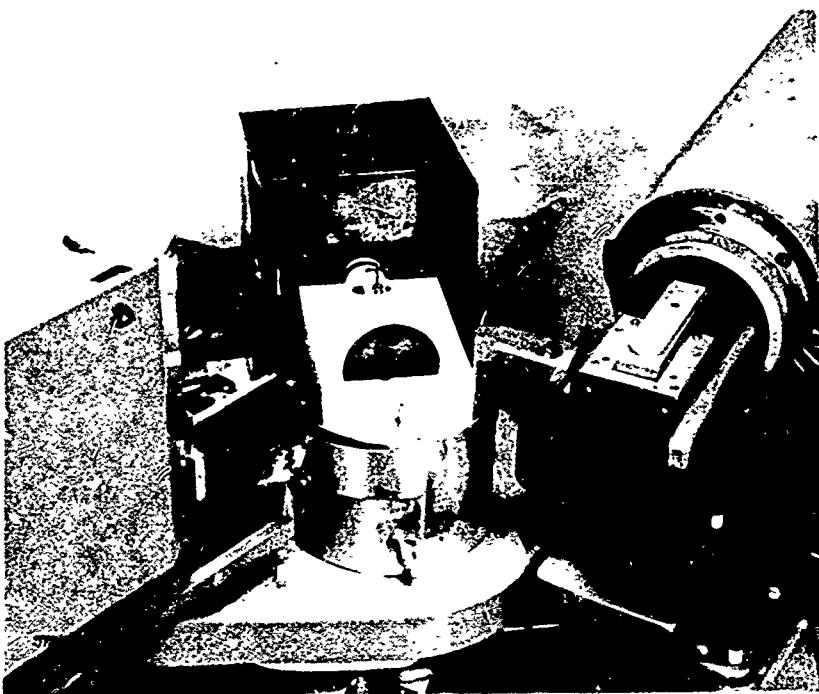


Fig. 2 - Cell mounted on spectrogoniometer. Part of the shielding was removed for this photograph.

More precise alignment may be obtained by having a light beam traverse from the detector to the specimen to the source slit, the reverse of the path traversed during registration of the diffraction patterns. Two standard detector slits are mounted inverted on the detector slit bracket using the holes and bolts that normally carry the detector and soller slits. A small light bulb placed between the detector tube housing and the first slit gives a collimated beam for alignment. The specimen surface is then brought into position so that the reflected slit image impinges on the x-ray source slit. When horizontal and vertical alignments are achieved, the slit image will not move during rotation of the specimen or in swinging the spectrogoniometer through its full arc, and the angle between the source and detector slits is bisected by the normal to the specimen surface. Following alignment, the desired detector and soller slits are placed in their

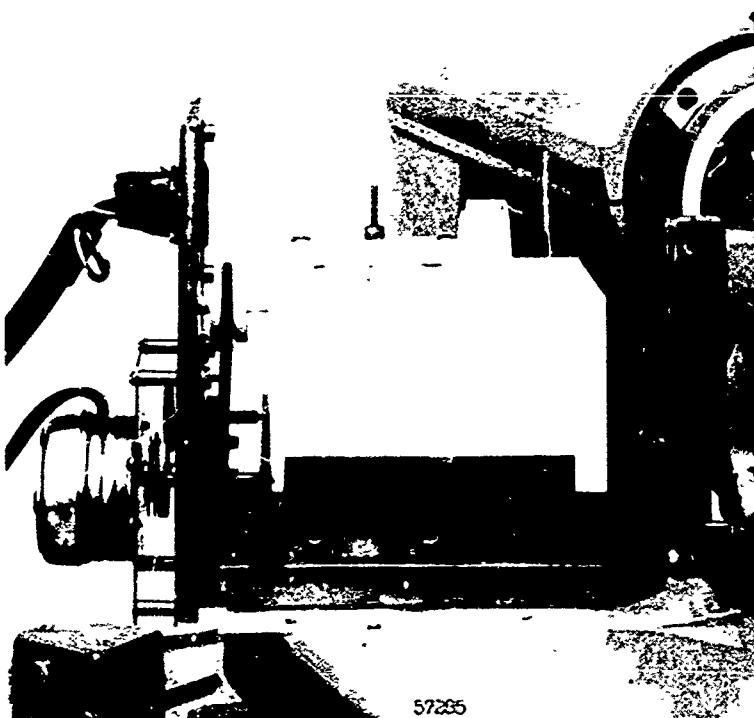


Fig. 3 - Cell mounted on spectrogoniometer. Part of the shielding was removed for this photograph.

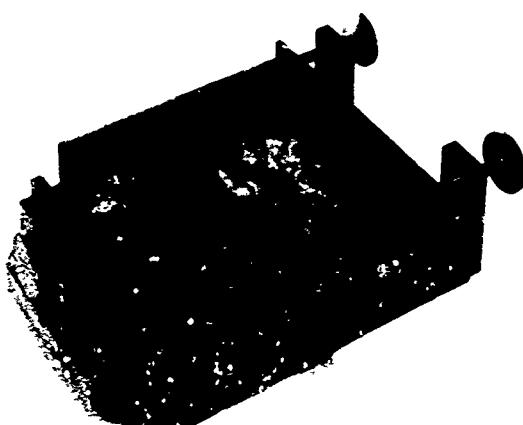


Fig. 4 - Cell mounting

usual positions and the cell is ready for use.

For studies requiring the absence of air, the cell may be enclosed in a flexible bag of plastic material to form a gas tunnel in which a positive pressure of the special atmosphere is maintained. Connections to the gas supply and to the source and detector slits may be made with elastic bands. Submersion heating or cooling coils may be placed in the electrolyte for varying or controlling temperature.

APPLICATION OF THIS CELL TO THE SILVER ELECTRODE IN KOH SOLUTION

Investigation of the silver electrode in KOH solution is of interest since silver oxides comprise the positive active material of charged silver-zinc and silver-cadmium storage batteries. It is desirable therefore to know the reactions and products occurring in alkaline solutions when electrodes of AgO , Ag_2O , or mixtures of the two are cathodically reduced, and when silver electrodes are anodically oxidized. Some confusion in investigations of the silver electrode has been caused by the report of Jones and Thirk (6) of what they believed was the x-ray diffraction pattern of AgO . The pattern reported has since been shown to be that of the so-called silver peroxysulfate $\text{Ag}_7\text{O}_8\text{SO}_4$ and not AgO (7). Biggs, Dugdale, and Wynne-Jones (2) confirmed that the final product of

anodizing Ag in H_2SO_4 was mainly the peroxy sulfide and not AgO . Jones and Thirsk (6) also reported finding traces of what could be a suboxide of silver mixed with Ag_2O but this was due to separating the true pattern for AgO into two patterns, one closely resembling the pattern of Ag_2O and the other resembling a face-centered cubic substance having $a_0 = 4.56\text{ \AA}$ (Ref. 7). The x-ray diffraction pattern for monoclinic AgO is well established now and has been given by Scatturin, Bellon, and Zannetti (8), and Graff and Stadelmaier (9) among others. Tetragonal AgO , having a diffraction pattern that resembles monoclinic AgO rather closely, was prepared by McKie and Clark (10) through the action of ozonized oxygen on hot silver powder. Electrochemically prepared AgO always has the monoclinic form, as far as is known.

In earlier work at this Laboratory, a silver electrode was removed from a cell following various amounts of charge or discharge and then examined by x-ray diffraction after rinsing the electrode in water and blotting dry (7). In alkaline electrolyte, the only materials identified were Ag, Ag_2O , and AgO . The presence of these materials and no others was verified by Dirkse (11) and Briggs, Dugdale, and Wynne-Jones (2).

The Ag sample (99.99+ percent purity) used for the present studies was a 57-mm-diameter, 0.25-mm-thick disk with an apparent working area of 21.9 sq. cm. Despite pressing the disk was not perfectly flat since it was cut from a roll of sheet silver. Consequently the recording of the x-ray diffraction pattern showed variations with sample rotation because the sample surface was not all in the same plane. The counter electrode was sintered Ag and the electrolyte was 20% KOH. The electrolyte was not protected from CO_2 in the atmosphere. Potentials were recorded against a Ag/Ag_2O reference electrode.

X-ray diffraction patterns of the Ag disk before any electrical or chemical treatment showed that the Ag was very highly oriented with the (110) plane lying in the surface of the sheet. After the potential had been at the Ag_2O/AgO plateau for 25 minutes at 4.8 ma during the initial charge (anodic oxidation), the diffraction pattern showed mainly AgO and a little Ag_2O . The oxide patterns had broad lines of low intensity indicating very poor crystallinity and/or small particle size. The oxides did not appear to exhibit any preferred orientation. Further patterns taken during the charge and after O_2 evolution were essentially the same. During the following discharge, the patterns indicated that poorly crystallized Ag_2O formed first, and then metallic Ag formed with about the same degree of orientation as originally found. The patterns of this Ag had slightly lower intensity suggesting the possible presence of a film formed on the base metal. This film might be an amorphous layer of Ag from the discharge that masks the underlying metal.

After the reduced sample stood without rotation for 16 hours, the part immersed in the electrolyte appeared rougher than the rest of the sample. An x-ray pattern of this rougher portion, taken with the sample stationary, showed a stronger development of (111) planes than there had been in previous patterns. This may indicate that spontaneous deposition from solution formed a preferred texture after standing in the electrolyte, or that a surface film recrystallized. The latter possibility is supported by the possible presence of an amorphous film immediately after discharge. There may be a connection between this and the fact that chargeability of Ag electrodes varies when they are allowed to remain discharged for several days (12).

There was often a secondary potential plateau while charging at the Ag/Ag_2O plateau. For example, at the 2- or 3-hour-charge rate there was usually a plateau at about 70 to 100 mv with respect to the Ag/Ag_2O reference after the first one at 25 to 50 mv. Ag_2O formed irregularly during a charge, being heaviest in lines that had been the liquid/air interface while the disk had been stopped for long times. The electrode potential usually began to vary with rotation shortly before changing from one potential plateau to another, due to irregular oxide coatings. Potentials became steady again after the next plateau was reached. This occurred both on charge and discharge. In general, oxide

coatings were heavier after the initial charge-discharge cycle. This was attributed to surface roughening.

After holding the potential at the final plateau for long periods of time (up to 41 hours), x-ray patterns still indicated only AgO of poor crystallinity. There were no diffraction peaks that could be attributed to a higher oxide. When the electrode was stationary, the pattern was stronger than when the electrode was rotating. In order to prevent Ag from plating onto the counter electrode, it was wrapped with three layers of cellulosic material of the type used as a separator in commercial silver-zinc storage batteries. The sample electrode was again held at the oxygen gassing plateau for several hours, but the AgO pattern was about the same as it had been before wrapping the counter electrode.

The final charge plateau of the rotating electrode was about 0.1 volt lower than normal for O_2 evolution on AgO. This lower gassing potential was an effect of rotating the electrode in and out of the electrolyte. Using a stationary electrode, the potential continued to rise for about 0.1 volt after the onset of O_2 evolution. There is evidence that O_2 forms through a peroxide mechanism in alkaline solution, probably by OH^- going first to O_2^- , which then goes to HO_2^- , and finally to O_2 . Although the electrode remained wet with electrolyte while it was out of the solution, all or almost all electrolysis probably took place on the portion of the electrode immersed in the solution, and an unstable ion such as O_2^- or HO_2^- could decompose instead of building up to the usual concentration. This resulted in the potential of the rotating electrode not rising as high as normal for a stationary electrode.

The last area of the disk to charge was the first area to discharge, judging by visual observations. The potential reached the lower or Ag_2O/Ag plateau when the electrode became covered with Ag_2O . X-ray diffraction at this point showed poorly crystallized Ag_2O and AgO. Metallic Ag appeared and gradually increased as a discharge continued at the lower potential plateau. At the time the electrode appeared visually to be about one-half metallic Ag, the diffraction patterns showed increasing Ag_2O and considerable AgO but Ag from the discharge could not be definitely identified. This may be due to poor crystallinity of the Ag. The x-ray pattern showed only metallic Ag at the end of a discharge and this Ag was not well developed. Even the strong line from the substrate (110) was obscured to a large degree.

The sample disk was abraded with a fine polishing cloth in an attempt to lessen the degree of orientation. The metallic Ag pattern showed much more random orientation after abrasion. Potentials measured during charges and discharges after abrading were similar to those obtained previously.

Figure 5 is an example of the type of diffraction pattern obtained after the sample was abraded. The electrode had been charged 17 hours at 2 ma (about the 6-hour rate) and then charged an additional 1.2 hours at 8 ma before beginning this pattern. Potential with respect to the Ag/Ag_2O reference varied from 505 to 530 mv. The electrode was rotating with an 8-ma current flowing during this pattern. Figure 5 shows a strong pattern for poorly crystallized AgO with the only other pattern that of metallic Ag. This Ag probably comes from the substrate since the Ag pattern is greatly obscured. The peaks have somewhat uneven contours because of lack of flatness of the specimen.

The diffraction pattern shown in Fig. 6 was made during the following discharge at 1 ma when the electrode appeared visually to be nearly covered with Ag_2O . The potential dropped from 160 mv to 109 mv with respect to the reference during the 25 minutes required to record this pattern. Figure 6 shows a pattern of residual AgO, a pattern for metallic Ag no stronger than at the end of the previous charge (Fig. 5), and a strong clear pattern for Ag_2O . This pattern of Ag_2O is noticeably stronger than one obtained after a long charge at the Ag_2O plateau where about 90% as much Ag_2O formed. At the end of the discharge, when the electrode was reduced to metallic Ag, the diffraction pattern showed

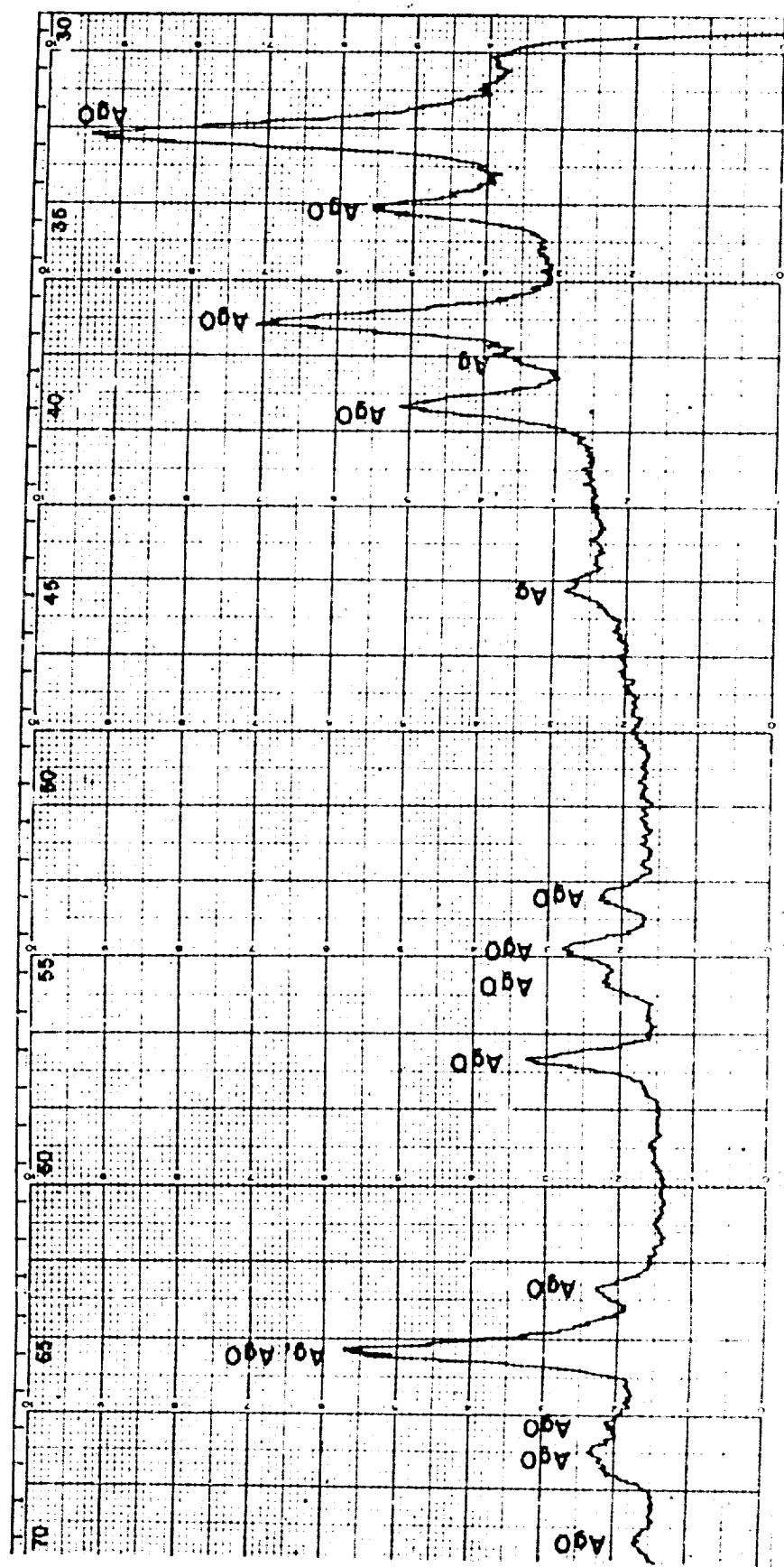


Fig. 5 - Spectrogoniometer CuK α x-ray diffraction pattern of abraded Ag after charging to O₂ evolution. Pattern made with sample rotating and current flowing. Numbers at top give degrees 2 θ .

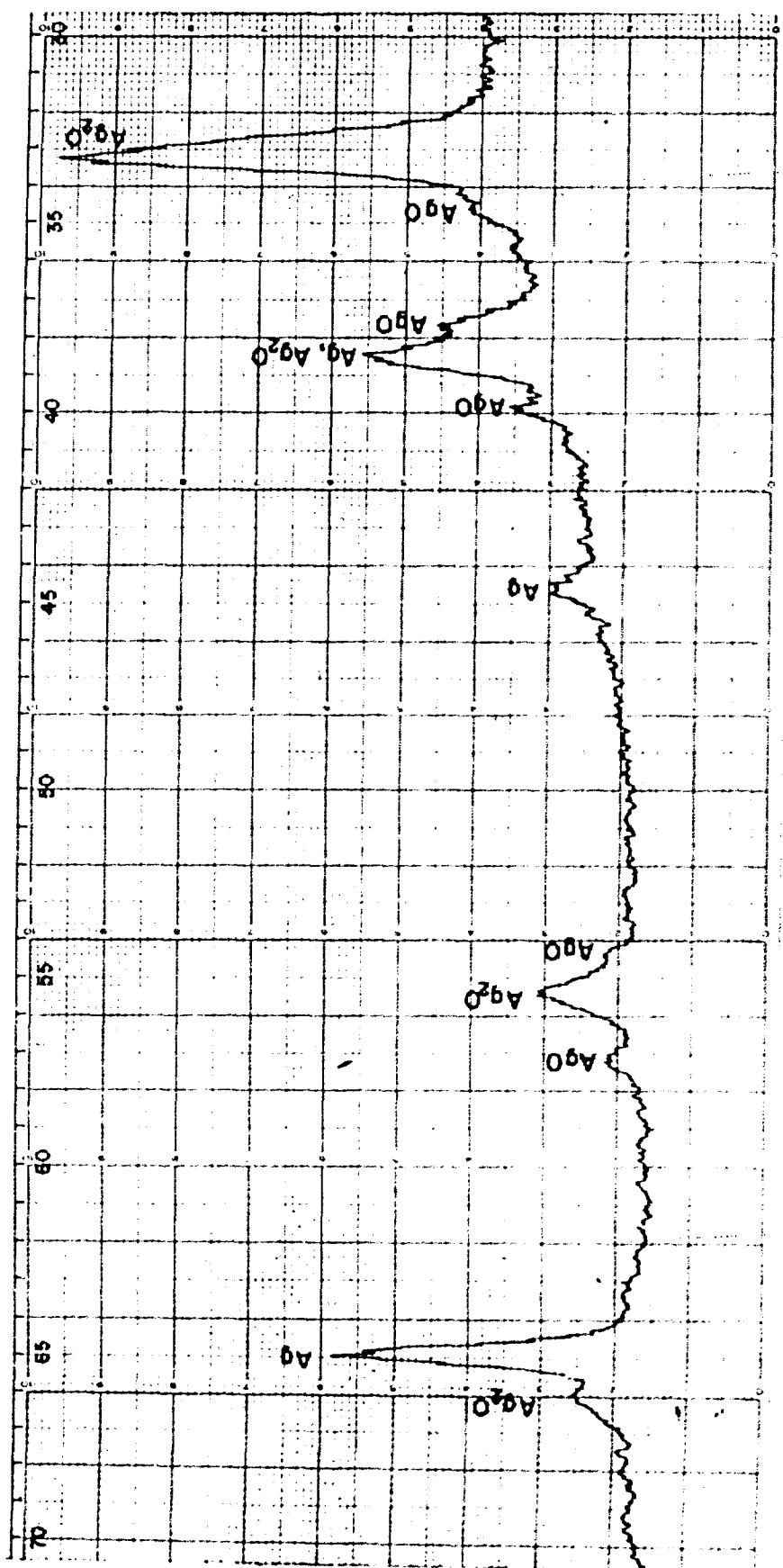


FIG. 6 - Spectrogoniometer CuK_{α} x-ray diffraction pattern of rotating sample during discharge as electrode became covered with Ag_2O . Numbers at top give degrees 2 θ .

polycrystalline metallic silver, but the peaks were not as strong as they had been in the first pattern recorded immediately after abrading the sample. Perhaps the abraded layer was being consumed and the metallic Ag from discharge was not being recrystallized fast enough to give a good pattern. The metallic Ag forming on discharge may require time in the electrolyte to grow from amorphous crystalloids. Future work will be done in an attempt to clarify this.

Cell capacity in these initial runs was irregular, being 4 to 5 ma-hr initially or after abrading, and then increasing to 10 to 16 ma-hr at charge currents of 1 or 2 ma. Higher currents resulted in less capacity. Discharge capacity was always less than the charge capacity. Some loss could be expected since it has been shown that efficiency in 20% KOH is under 100% due to gradual loss of Ag (13). The efficiency obtained with the experimental arrangement described here was usually about 70% to 80%, which is considerably worse than the 0.5% loss described earlier (13). It is assumed that most if not all of this loss was due to Ag going into solution. During a charge some black particles precipitated in the electrolyte, although the electrolyte remained clear. Sometimes the electrolyte was a yellow or tan color by the end of a discharge but the color soon disappeared. The electrolyte became particularly dark in the first discharge after wrapping the counter electrode. This probably was due to reduction of dissolved Ag ions. The precipitated material that formed after standing discharged overnight was identified as metallic Ag.

The preliminary work on the silver electrode has shown that this new electrolytic cell for x-ray diffraction studies is practical and easy to use once the equipment has been set up. Patterns are improved over those shown in Figs. 5 and 6 when the sample is sufficiently rigid and flat. An increased speed of rotation will minimize both unevenness in the diffraction pattern due to lack of sample flatness and decomposition of any unstable material formed during electrolysis.

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